

Methyl Bromide Concentrations in Air Near Fumigated Single-Family Houses

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by

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Abstract

Concentrations of methyl bromide in air were measured in close proximity to a single-family house during the treatment phase of each of seven fumigations. Air monitoring stations were located at 10 feet from the fumigated structure. Indoor monitoring stations in neighboring houses were located in the rooms closest to the fumigated structure. Monitoring was conducted during the approximately 24-hour treatment phase of the fumigation. Airborne methyl bromide levels at 10 feet from the house often exceeded the 210 ppb target level of the California Department of Pesticide Regulation. These outdoor levels ranged from less than 0.019 to 1.495 ppm. About 60 percent of the valid samples were below 210 ppm while about 40 percent were above. The majority (95% of all samples collected) of air levels inside neighboring houses were far below the 210 ppb level, with a range from non-detected (0.012 ppm) to 0.351 ppm.

Introduction

The single-family dwelling house is the most typical site of fumigation of non-commercial structures. Little information is available on offsite migration of fumigant gas during the treatment and aeration phases of fumigation of single-family houses with methyl bromide (bromomethane, CAS #74-83-9) or other fumigants. Unpublished data from the Department of Pesticide Regulation (DPR), Worker Health and Safety Branch, indicated that there were measurable levels of fumigant gas up to 15 feet away from structures both during treatment and aeration. DPR is concerned about the magnitude of airborne methyl bromide levels in the vicinity of fumigated single-family dwellings, in light of new toxicology data received for this fumigant. DPR is now recommending (target exposure value) airborne exposure not exceed 210 parts per billion (ppb) averaged over 24 hours (State of California Memorandum, Nelson to Wells, 1992). This study was designed to determine methyl bromide concentration during the treatment phase of fumigation of single-family dwellings following current work-practices. The concentration versus distance relationship will be compared to DPR's target exposure value. Results of this study may be used to determine if there is presently adequate control of fugitive emissions during fumigation to prevent exposure to persons in the vicinity above the DPR target exposure value. Additional impetus for this study came from the listing of methyl bromide as a Proposition 65 chemical when used for structural fumigation. This listing imposes additional restrictions on the fumigant when used in California. For reference, methyl bromide product labels require the use of respiratory protection if workplace levels exceed 5 parts per million (ppm) and the current occupational exposure limit value of Cal/OSHA for methyl bromide is 5 ppm, averaged over an 8 hour workday. The purpose of this monitoring was to characterize air levels of methyl bromide near structures during the treatment phase of fumigation. This study also measured air levels inside neighboring structures.

This study was conducted as part of a much larger study designed to examine the magnitude of downwind levels of methyl bromide during the aeration of fumigated single-family structures. The downwind levels associated with aeration are summarized in Worker Health and Safety Branch Report HS-1713.

Methods and Materials

Test Site

The study was conducted in decommissioned base housing at the former Mather Air Force Base in Sacramento, California. All housing in this area was vacant and the grounds were secured by a security service furnished by the base, providing ideal conditions for extensive monitoring of methyl bromide offsite movement. One house was selected as the test house. The same house was fumigated in every test. The test house had approximately 2,590 ft² of floor area including the garage area, and an inside volume of approximately 20,700 ft³. Five surrounding houses (one on each side, two behind and one across the street from the test house) were selected for indoor sampling (See Appendix A1). The two houses in the back (the north side) were on a slight elevation (approximately 1 meter) relative to the fumigated test house. All the houses were one-story three bedroom two bathroom of a standard ranch-style tract construction, using slab foundations. All had attached garages which were not accessible from within the house. All had been vacant for less than a year and were in good repair, unfurnished, and had no obvious structural defects. The same semi-permanent monitoring stations, set up around the test house and inside the neighboring houses, were used in each test. The open spaces surrounding the houses were not divided by any fences. This common area was covered in low-lying vegetation with some trees present. The site is diagrammed in Appendices A1 (General Overview) and A2 (Detail Map).

Fumigation Procedure

A commercial fumigation company in Sacramento was contracted to perform the fumigations. Each fumigation was performed by a two- or three-man crew. The crew deployed industry-standard tarpaulins to enclose the structure, using sand-filled canvas tubes ("sand snakes") to form a seal against the soil. Tarpaulins were joined by rolling the edges together, then clamping the rolls with steel clips. After sealing the tarps, the crew would set-up the injection system, consisting of the 150 pound methyl bromide tank (Meth-O-Gas, 99.5% methyl bromide, 0.5% chloropicrin as a warning agent), a high-pressure hose connected to a propane-powered water-heater (to

warm the gas) and an injector hose into the house. The fumigant was applied at a rate of 3 lb./1000 ft³, which is the upper limit of the label rate and typical of treatments in Northern California. It usually took about 25 minutes to inject the 62 pounds of methyl bromide (20,700 ft³ x 3 lb/1,000 ft³). After injection, the crew would post the required warning signs, dismantle the injection equipment and leave.

All fumigations were conducted in the morning, before 1200 hours. The series of fumigations began in winter, with the seventh and final fumigation occurring in early spring.

Aeration Procedure

The morning after the fumigation (between 22 and 24 hours post-application), the fumigation crew would return and begin aeration. Just prior to the start of the aeration procedure, a confirming measurement to determine the concentration remaining within the fumigated structure was obtained with a Fumiscope[®] (measuring ounces per 1,000 ft³). Two methods were used for aeration, both ultimately purging the structure of methyl bromide residue. The aeration procedure had no effect on the monitoring during the treatment phase of the fumigation and is therefore not detailed in this report. A report concerning the environmental effects of aeration is available (Worker Health and Safety Report HS-1713).

Sampling Media

To monitor methyl bromide down to the low level which may be encountered near tarpaulin-covered structural fumigation, sampling followed the method of the National Institute for Occupational Safety and Health (NIOSH method #2520) and used petroleum-based charcoal tubes (SKC-West, Inc., Fullerton, CA 92634, catalog # 226-38-02). This sampling medium consists of adsorbent contained in two sections, a primary tube containing 400 mg of charcoal and a secondary (backup) tube containing 200 mg of charcoal. During sampling, these two sections were connected with a short piece of plastic tubing (TYGON[®] or equivalent). To avoid breakthrough associated with collection of methyl bromide on charcoal, all samplers were calibrated to draw no more than 10 to 12 liters of air through each set of sampling tubes in a sample period. The flow rates varied according to the sampling characteristics (piston displacement, motor speed) of the pumps. Actual volumes were calculated from each pump's unique conditions.

Indoor Monitoring

Each of the five houses neighboring the fumigated test house was assigned an identification number equivalent to its address number (see Appendix A2). Houses 135, 141, and 138 were within about 50 feet of the fumigated house. Houses 107 and 109 were within 100+ feet of the fumigated house. Within each house, the room closest to the test house was selected as the sampling room. In Houses 138 and 107, these were bedrooms; in 109 it was the living room; in 135 it was the master bedroom and in 141 it was the dining area. All samplers were situated next to a closed window (single pane, aluminum frame). Each sampling site consisted of one tripod (to elevate the sampling media to 4 to 5 feet above the floor), one sampling pump (MSA Model C-210 Portable Pump [No.468200]), one charger unit for long-term powering of the pump (MSA Model 463679) and the sampling train (media and necessary tubing). These samplers were operated with house power. All doors and windows were kept closed, with only intermittent front door opening to replace sampling media. Between tests, samples were collected from within the treated house and these neighboring houses to ensure the fumigant had dissipated.

Exterior Monitoring

Exterior sampling sites were located on all sides of the structure. Samplers were placed at 10 feet from the outer surface of the tarpaulin. During the first test, three samplers were also placed at 50 feet. However, these sites yielded no detectable level results. Because of equipment allocation constraints, these sites were dropped from subsequent sampling. Each sampling site consisted of one metal stake (to elevate the sampling media to 4 to 5 feet above the ground), and the same MSA Model C-210 Portable Pump and sampling media set-up used for interior monitoring. Because the length of sampling time required more power than the internal batteries could provide, supplemental battery packs were designed for the MSA units. The added power supply allowed sampling to continue beyond the normal 6 to 8 hours provided by the internal batteries. External batteries were replaced as necessary to ensure constant power to the pumps.

Each sampling site was assigned a unique location identification site number between 1 and 8. Site locations are shown in Appendix A1.

Sampling Schedule

Sampling sites were readied by placing the necessary air samplers and sampling media on the stakes/tripods before the fumigation crew began their tarping of the house. After the tarping was completed, the sampling stations were rechecked to verify that none of the equipment had been disturbed during the tarping procedure. The fumigation crew was notified when all sampling stations were verified. The fumigation crew would then begin the injection of the gas. A few minutes later the sampling equipment was activated.

After complete injection of the gas, the fumigation crew packed their injection equipment, posted the fumigation site with warning signs and left the area. Pumps were again checked for operation and then the sampling crew left the equipment to run overnight. All pumps were normally calibrated to run for approximately 21 to 24 hours. During Test Two, samplers were set to run for 12 hours and then have their sampling media changed. After assessment of the data generated from that test, and because of general logistic problems involved in sending personnel out late at night, all subsequent samples were taken on an overnight schedule.

During collection of the charcoal tubes after sampling, any unusual conditions (sampler failure, battery failure, tube dislodgment, etc.) were noted and reported to the sample processing manager (see Sample Storage and Analysis).

Sample Storage and Analysis

After completion of the sampling period, the charcoal sampling tubes were returned to the base station (garage of House 138) and given to the sample processing manager for check-in and preparation for storage. After logging in the sample number, tubes were separated (primary "A" tube from secondary backup "B" tube) and capped. Capped tubes were then placed on dry ice and stored until delivered to a freezer (temperature -20 °C). After all tubes were collected from a test period, the tubes were taken to Chemistry Laboratory Services of the California Department of Food and Agriculture (CLS/CDFA) for analysis of methyl bromide. The methodology used in analysis is given in Appendix B. The time between sample collection and final analysis varied from 1 to 4 weeks. Results were reported in micrograms per sampling tube. In cases where there were detectable amounts on the secondary tube, the amount quantified on the secondary tube was combined with the amount determined on the primary tube. If the backup value exceeded 25% of the primary, the sample was considered void. In a few cases, there was evidence that the tubes had been incorrectly attached to the pumps, i.e., reverse order. In such cases, it was fairly obvious (detectable levels in the "B" tube, non-detectable levels in the "A" tube) and these tubes were not considered void.

Weather Monitoring

Local temperature and relative humidity within the houses and at the 10 foot sampling sites were measured using a hand-held meter (HANNA Instruments, Model HI 8564).

GLP Compliance

This study was not conducted under compliance with the Good Laboratory Practice standards (40 CFR 160) of the US Environmental Protection Agency. Deviations and/or amendments to the protocol were documented and are available in the raw data archives.

Quality Control

Quality control (QC) tests were conducted by the analytical laboratory (CLS/CDFA) to ensure accurate analytical results. These tests are the same as those reported on for the other larger aeration study conducted at the same time (See HS-1713). The field samples were analyzed at the laboratory in 12 batches. A set of three QC spikes, prepared in the laboratory, were analyzed with each batch. Each QC set consisted of a high- (8.52 ug), medium- (4.26 ug) and low-level (0.85 ug) spike. Spike levels were chosen to bracket expected field levels. Four additional sets were analyzed independently. In these sets the high, medium and low spike levels were 8.52, 2.26, and 1.13 ug, respectively. All 16 sets were combined for statistical analysis. Analytical recovery in these sets averaged 71.4 % (range 49-102 %). Percent recovery was significantly lower in high level spikes than in low level spikes.

To examine storage stability, twenty high- (14.2 ug) and twenty low-level (1.12 ug) spikes were prepared and five of each level were analyzed after 1, 2, 3 and 4 weeks of storage. There was evidence of loss of methyl bromide from low level spikes after one week of storage following spiking, but not from the high level spikes. There appeared to be no further loss at 2, 3 or 4 weeks of storage at either level.

Both the QC spikes and the storage stability spikes showed cyclical trends in recovery. Statistical analysis of the QC data is described in Appendix C. The uncertainty in sampling and analytical methods for methyl bromide suggests that the raw data not be modified for recovery or storage loss.

Retention of Raw Data

The testing agency (DPR/WH&S) will retain copies of all raw data for a minimum of 5 years. All raw data, protocol amendments, analysis requests/chains of custody and related paperwork will be retained.

Results

Methyl bromide concentration in air inside the fumigated house (Fumiscope[®] readings) just prior to the start of aeration averaged 20 ounces per 1,000 cubic feet (5190 ppm). This correlates to about 42 percent of the application rate of 48 ounces per 1,000 cubic feet (12,378 ppm). The concentration ranged from 3600 ppm to 7460 ppm in 6 tests (one test not measured), 22 to 24 hours following initiation of fumigation and immediately before tarpaulin removal. Concentrations within the fumigated structure were not measured immediately following introduction of fumigant.

The raw analytical results for airborne methyl bromide levels outside the fumigated structure and in neighboring houses are shown in Tables D-One and D-Two of Appendix D. The results from Sites 12, 13, and 14 used in Test One were all non-detectable (<0.012 ppm) and are not reported in the tables. Throughout the study, temperatures within the structures ranged from the low 50's to the low 70's. Relative Humidity varied between 40 and 65 percent. All pre-application, background samples indicated non-detectable methyl bromide levels in all structures.

Table I shows a summary of all valid samples collected at the 10 foot distance around the fumigated house. Approximately one-third of these samples showed levels above 210 ppb.

Table I
Methyl Bromide Concentration in Outdoor Air at Ten Feet
from the Fumigated House During Fumigation
(in ppm)

NUMBER OF SAMPLERS	MINIMUM	MEDIAN	MEAN *	95 TH PERCENTILE	MAXIMUM
44	0.019	0.188	0.261	0.665	1.495

*arithmetic

Table II shows a summary of all the samples collected from within the neighboring houses. House 135, 141 and 138 were within about 50 feet of the treated house. Houses 107 and 109 were 100+ feet from the treated house. Although there was a distance factor, due to the limited number of measurable values obtained, all data were combined. House 135 is treated separately below due to a non-standard sewer connection found to exist between this house and the fumigated house and discovery of empty drain traps in the treated house and in some of the test houses. This sewer connection may have directly introduced methyl bromide into the bathroom of the adjoining bedroom where the sampling equipment was located. A mean concentration was not calculated because of the large number of non-detected values. Non-detected values were calculated using the minimum detectable level reported by the laboratory (0.5 µg/sample) and dividing by the volume of air sampled. The default sample volume of 10 liters yields an minimum detectable level of 0.012 ppm. Sixty percent of all the interior samples were non-detectable.

Table II
Methyl Bromide Concentration in Air Within
Neighboring Houses During Fumigation
(in ppm)

	NUMBER OF SAMPLERS †	MINIMUM	MEDIAN	95 TH PERCENTILE	MAXIMUM
All Houses	34	ND	ND	0.203	0.351
House 135‡	7	ND	0.035	0.351	0.351
All But House 135	27	ND	ND	0.081	0.203

†Based on seven tests in each of five houses (one observation missing)

‡House with faulty sewer connection

ND=0.5µg (c.a. 0.012 ppm)

Discussion

DPR considers 210 ppb (0.21 ppm) to be a target exposure control level for methyl bromide. This level is measured as a 24-hour average air concentration. The outdoor results show this level was routinely exceeded within the 10 foot distance surrounding the fumigated structure. The mean and 95TH percentile values are comparable to values obtained from monitoring at the same 10-foot distance during the subsequent aeration phase of these fumigations (See WH&S HS-Report 1713). This observation is not unreasonable in light of the observation that at the beginning of the aeration phase less than half of the calculated initial amount of fumigant remained.

The site with the greatest number of values exceeding 210 ppb was Site 3. This site was located in the area bounded by the house on one side and the attached garage on the adjoining right-angle side. This effect, of being flanked on two sides by the fumigated structure, and somewhat sheltered by the structure from wind, may have

contributed to the large number of >210 ppb values being found there. One sample from this site registered a value of 1.5 ppm (1,495 ppb), the highest single result during any of the tests. This observation indicates that local topographic features may have a large influence on nearby airborne levels of fumigant during fumigation. These features would include flanking walls of the fumigated structure, concrete or tightly spaced wood enclosure walls, extremely close ("zero-lot line") neighboring houses, semi-enclosed patio structures, etc. Of all outdoor samples collected, 27 (54%) were below the 210 ppb target level, 18 (36%) were above and 5 (10%) were voided samples (primarily from pump failure).

For the samples collected within neighboring houses, the highest value was 351 ppb. This value was measured in House 135. Two factors were thought to be responsible for the higher levels measured in this house. House 135 and the fumigated house were found to share an uncommon sewer connection. The sewer lines from these houses were directed to a midpoint between the houses where they joined a single drain line. This line then ran down to the sewer main under the street. This non-standard connection formed a direct line from the fumigated house to House 135. In addition, some drain traps in the fumigated house and some of the test houses (including 135) were found to be empty. Any empty trap would allow methyl bromide laden air out of the fumigated house and into House 135 via the sewer system and any empty trap in House 135. In House 135, a shower in a bathroom opening to the room being monitored was found to be emitting methyl bromide into the house. This condition was unique to these houses. All other test houses were not thought to be affected by empty drain traps nor by direct sewer connection. By Test 7, the problem had been recognized and solved by adding a low-volatility, glycol-based fluid to all water traps in all test structures. This fluid was used by the base management to both prevent pipe damage from freezing and to prevent infiltration of sewer gases into the vacant homes. Discounting values from House 135 (except for Test 7), there were no other values exceeding 210 ppb. As shown in Table II, the effect that House 135 has on the 95th percentile for all houses can be rather large. With House 135 included in the data, the 95th percentile is 0.203 ppm. Excluding House 135 from the data set reduces the 95th percentile to 0.081 ppm. There was one other site of unusual results: House 109, during Test 2, had a value of 203 ppb. This anomalous value, 2X to 3X higher than any other values during any other test, cannot be explained. Outdoor samplers that could have potentially been upwind of House 109 showed a highest value of 557 ppb. It would be difficult to explain a mechanism by which an outside air level of 557 ppb at 10 feet could result in an indoor level, more than 100 feet away, of 203 ppb. This result may be from abnormal (and undetected) sampling pump behavior or from undetected laboratory error. The majority of samples from within houses were either non-detectable (21 samples, 60%) or were at levels below the DPR target value (12 samples, 35%). Only one interior sample was above 210 ppm (351 ppb, 2.5%) and one sample was lost from pump failure (2.5%).

Conclusions

The final analysis of these data tends to support the following conclusions regarding the treatment phase and resulting environmental levels of airborne methyl bromide:

1. During the treatment phase of fumigation, 24-hour average outdoor airborne levels of methyl bromide may exceed 210 ppb within 10 feet of a fumigated structure.
2. During the treatment phase of fumigation, airborne levels of methyl bromide within neighboring structures greater than 50 feet away will probably not exceed 210 ppb as a 24-hour average.
3. Local topography of the area surrounding a fumigated structure may contribute to higher levels from "pockets" of fumigant accumulating in areas of restricted wind/air flow.
4. Fumigant may travel through the sewer system to neighboring houses if the drain water-traps are not filled with liquid.

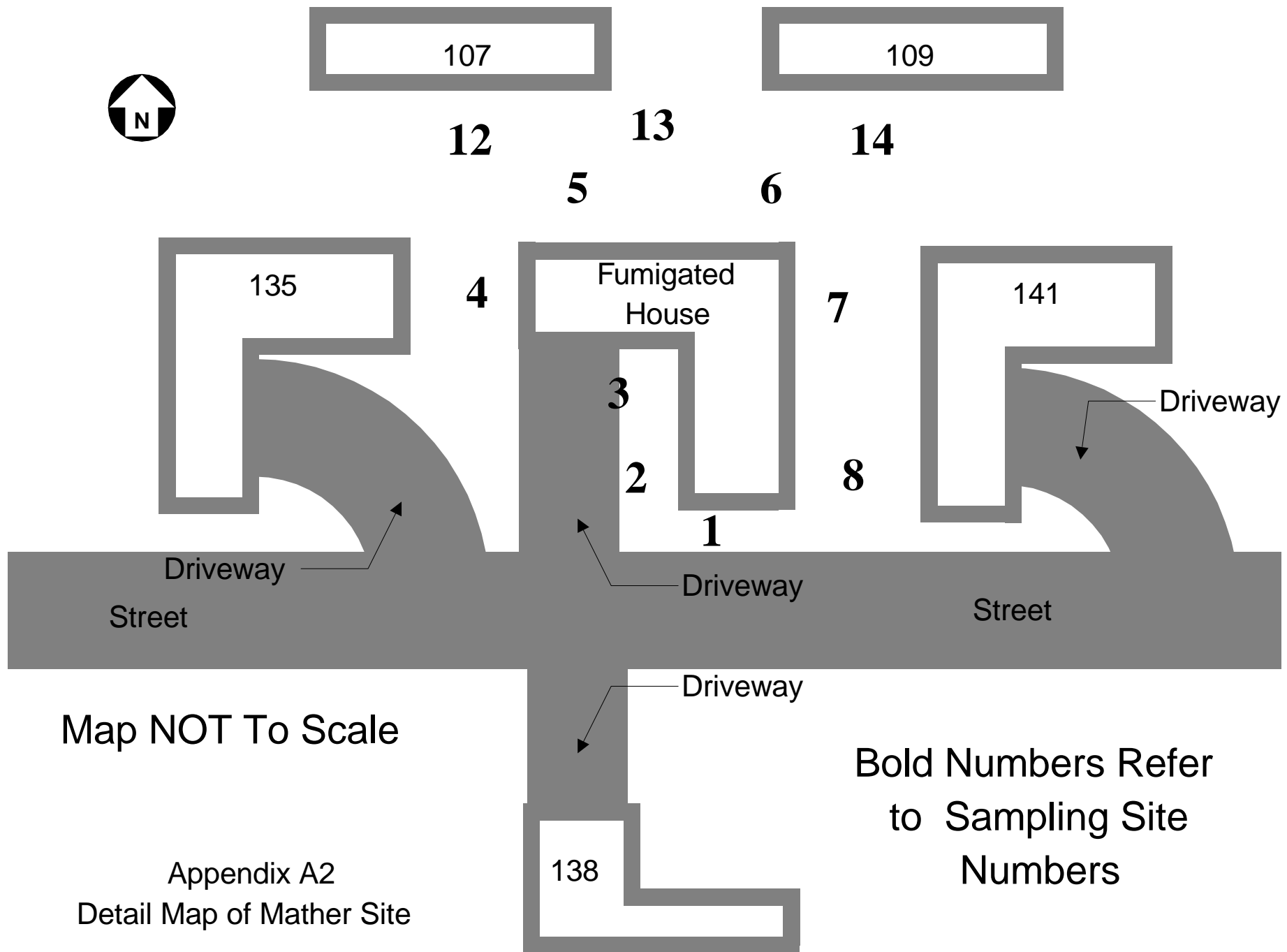
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Disclaimer

Use or mention of specific products or trade-names in this report is in no way an endorsement of such products or trade-names by Cal/EPA, Department of Pesticide Regulation or the State of California, nor is criticism implied of similar products not mentioned.





Appendix B

Laboratory Analytical Method for Methyl Bromide

Scope

This method is for the determination of methyl bromide in charcoal tubes.

Principle

Methyl bromide is extracted from the charcoal tube with ethyl acetate. Analysis is by gas chromatography equipped with electron capture detector.

Reagent and Equipment

Ethyl acetate, Reagent grade, purity checked prior to use.

Vial, 5 ml, white cap

Miscellaneous glassware

Standard Preparation

Ethyl acetate is added to a calibrated 100 ml volumetric flask to the calibration line and the weight taken. Then about 3 ml of solvent is taken from the flask. This flask is connected by a Luer lock needle to a cylinder containing 99.5% methyl bromide, calibrated to a flow rate of about 50 ml/min. The gas is allowed to bubble into the solvent for about 1 minute. The needle is then removed from the ethyl acetate and the gas turned off, in that order. The volumetric flask is stoppered and allowed to equilibrate at ambient temperature. It is then filled to the calibration line. The weight difference between the flask filled with solvent and the flask after gas bubbling is the amount of the methyl bromide. This is the primary stock solution. It is kept in sealed 2 ml ampoules in a freezer for storage. From this solution the following working standard solutions are prepared: 0.28, 0.56, 1.01, 1.98 and 3.96 ng/ μ l.

Spike Preparation

The charcoal tube is broken in the middle. Spiking solutions, which are made from the primary stock solution, are introduced into the tube by a 10 μ l syringe. Levels of spike are 0.85, 4.26 and 8.52 μ g per sample.

Analysis

The charcoal in the tube is put into a white cap vial containing 4 ml of ethyl acetate. The vial is shaken gently and allowed to settle for two hours. The extract is ready for analysis.

Equipment Conditions

Gas chromatograph: Hewlett-Packard 5880A with Hewlett-Packard 7672A Automatic sampler

Injection volume: 2 μ l

Column: J & W DB-625 30 m \times 0.53 mm \times 0.2 μ m

Temperature profile:

Initial value: 40 $^{\circ}$ C

Initial time: 2.5 minutes

Program rate: 30 $^{\circ}$ C/min

Final value: 200 $^{\circ}$ C

Final time: 3 minutes

Injector temperature: 250 $^{\circ}$ C

Detector temperature: 350 $^{\circ}$ C

Gas flow:

Helium (carrier): 28 ml/min

Argon-Methane: 42 ml/min

Retention Time: 0.64 min

Calculation

μ g methyl bromide/sample = [(std, ng/ μ l)(pk ht sample)(μ l std)(vol of solvent, ml)]/(pk ht std)(μ l sample)

Recovery

60% to 90%

Detection Limit

0.5 μ g/sample

Appendix C

Statistical Analysis

Quality Control Data

Analytical Recovery

A set of three QC spikes was included with each batch of field samples analyzed. Each set consisted of a high- (8.52 µg), medium- (4.26 µg) and low-level (0.85 µg) spike. In addition to the 12 spike sets analyzed with the field samples, four sets were analyzed independently. In these sets the high, medium and low spike levels were 8.52, 2.26, and 1.13 µg, respectively. Recovery at each spike level is shown in Table C-One.

Table C-One
Percent Analytical Recovery in QC Spikes

Type	Spike Level	n	Mean	Coefficient of variation
<hr/>				
With field samples	<i>High</i>	12	61	11
	<i>Medium</i>	12	72	16
	<i>Low</i>	12	80	10
Independent	<i>High</i>	4	82	20
	<i>Medium</i>	4	69	19
	<i>Low</i>	4	67	7
<hr/>				
Combined	<i>High</i>	16	66	19
	<i>Medium</i>	16	71	16
	<i>Low</i>	16	77	12

The 16 sets of spikes were combined for statistical analysis. Analysis of variance (randomized blocks model using sets as the blocks) indicated there were significant differences among spike levels in mean recovery ($F=4.15$; $df=2,30$; $p=0.0257$). Recovery was significantly greater at the low level than at the high level, while the medium level did not differ significantly from either of the others (Bonferroni t-tests with overall $\alpha=0.05$). Differences among the sets did not reach statistical significance ($F=1.81$; $df=15,30$; $p=0.0805$).

Storage Stability

Twenty high- (14.2 µg) and twenty low-level (1.12 µg) spikes were prepared and five of each level were analyzed after 1, 2, 3 and 4 weeks of storage. Recovery at each time is shown in Table C-Two.

Table C-Two
Mean Percent Recovery in Storage Stability Spikes

Spike Level	Weeks of Storage			
	1	2	3	4

High	70† (7)‡	66 (5)	62 (5)	67 (8)
Low	61 (9)	53 (9)	52 (4)	56 (6)
Combined	65 (10)	59 (14)	57 (10)	62 (12)

† For each mean n=5.

‡ Coefficient of variation

Analysis of variance (Week x Spike Level complete factorial model) showed significant differences among weeks ($F=7.16$; $df=3,32$; $p=0.0008$) and among spike levels ($F=72.39$; $df=1,32$; $p=0.0001$). The interaction was not statistically significant ($F=0.61$; $df=3,32$; $p=0.6151$), which means that while overall recovery was significantly higher in the high level spikes, the change in recovery from 1 to 4 weeks did not differ for high and low level spikes. Means comparisons (Bonferroni t-tests with overall $\alpha=0.05$) indicated that recovery for the combined spike levels was significantly lower after 2 weeks of storage than after 1 week. Week 3 recovery was not significantly lower than Week 2 (and was significantly lower than Week 1). Mean recovery after 4 weeks was higher than after 2 or 3 weeks, and did not differ significantly from any of the other weeks. However, this apparent decreasing then increasing trend probably represents variability inherent in recovery rather than a real effect of storage time. If recoveries were plotted chronologically for all of the QC spike sets, it would be seen that the week-to-week variation in storage stability samples is well within the range of set-to-set variation in the QC spikes. Moreover, the QC spike sets exhibit cyclical trends in recovery, even though they were all analyzed with no storage interval. (These cycles may indicate a need for better control over the analytical process.)

Although there appears to be no real loss of material between 1 and 4 weeks in storage, it does appear that there is loss between 0 and 1 week in the low-level spikes. Recoveries for high-level spikes were similar in QC and storage spikes, but with low-level spikes the recoveries were lower in the storage spikes, suggesting loss between Week 0 (when the QC spikes were analyzed) and the subsequent weeks. Mean recovery in the pooled storage stability spikes was compared to the mean of the pooled QC spikes at each level using t-tests for independent samples with unequal variances. For the low level spikes, recovery was significantly lower in the stored samples ($t=8.18$, $df=22.2$, $p=.0001$). These comparisons are shown in Table C-Three.

Table C-Three

Mean Percent Recovery in Storage Stability vs. QC Spikes

Spike Level	Type	n	Mean	Standard Deviation
High	QC	16	66	13
	Storage	20	66	5
Low	QC	16	77 ^{***}	9
	Storage	20	55 ^{***}	5

^{***} Significantly different; $p < 0.001$.

Appendix D Raw Data Tables

Table D-One

Outdoor Methyl Bromide Levels During Fumigation
10 feet from Fumigated Structure
(ppm)

Site Num.	Test One	Test Two	Test Three	Test Four	Test Five	Test Six	Test Seven
1	0.041	0.021	0.269	0.197	0.161	0.209	
2			0.665	0.159	0.145	0.108	0.063
3	0.236	0.557	0.081	0.437	1.495	0.978	0.552
4	0.048	0.125	0.490	0.112	0.287	0.106	0.318
5	0.045	0.019	0.345	0.247	0.121	0.193	0.183
6		0.155	0.091	0.062	0.200	0.142	0.281
7	0.085	0.318	0.178	0.236	0.323	0.405	
8		0.164					

Blanks are from non-sampled or failed pump sites.

Bold type indicates highest value for each test period.

Table D-Two

Methyl Bromide Levels During Fumigation
Inside Neighboring Houses
(ppm)

Site Num.	Test One	Test Two	Test Three	Test Four	Test Five	Test Six	Test Seven
107	<u>0.016</u>	0.020	<u>0.027</u>	<u>0.026</u>	<u>0.022</u>	<u>0.027</u>	<u>0.017</u>
109	<u>0.012</u>	0.203		<u>0.015</u>	<u>0.013</u>	<u>0.013</u>	<u>0.020</u>
135	0.351	0.067	0.035	<u>0.018</u>	0.067	<u>0.018</u>	<u>0.021</u>
138	<u>0.012</u>	<u>0.020</u>	0.024	<u>0.018</u>	<u>0.015</u>	<u>0.013</u>	<u>0.021</u>
141	0.020	0.051	0.081	0.046	0.043	0.039	<u>0.020</u>

Note: Values in underlined italic are based on Minimum Detectable Limit (MDL).

Blank is from failed pump site.